



Naval Fuels & Lubricants

Cross Functional Team

Qualification Report

Joint Oil Analysis Program Spectrometer Standards VHG Labs Inc. Qualification Report For D19-XXX Series Standards

NF&LCFT REPORT 441/13-015

4 November 2013

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NAVAIR Public Release 2013-1060

Distribution Statement A - Approved for public release; distribution is unlimited

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EXECUTIVE SUMMARY

The Joint Oil Analysis Program recently revised the specification for their spectrometric oil standards in order to incorporate an ICP-AES (Inductively Coupled Plasma Atomic Emission Spectroscopy) test method and to transition to commercially manufactured spectrometric oil standards. Previously a Rotrode-AES (Atomic Emission Spectrometry) test method was the only elemental test method used to verify the quality of the spectrometric oil standards. The Rotrode-AES test method was a labor and time intensive process that did not exhibit a high level of accuracy and repeatability. The JOAP wanted to incorporate an ICP-AES test method in order to obtain a more accurate determination of the true concentration of the elements in the spectrometric oil standards and to reduce the cost and time needed to conduct quality assurance. Both the Rotrode-AES and ICP-AES test methods are now part of the qualification inspection in order to ensure that the formulation of the commercially manufactured spectrometric oil standards is correct.

The JOAP D19 reference series has historically consisted of true gravimetric spectrometric oil standards. The formulation of the JOAP D19 reference series has remained constant throughout the history of the program in order to ensure the accuracy of the aeronautical and non-aeronautical limits in the JOAP Manual (NAVAIR 17-15-50.3 and 17-15-50.4). The importance of maintaining the formulation of the JOAP D19 reference series provided another reason for the JOAP to transition to commercially manufactured spectrometric oil standards. Commercially manufactured spectrometric oil standards are generally traceable to NIST Standard Reference Materials (SRM). This traceability further ensures that the formulation of the commercially manufactured spectrometric oil standards is correct.

This report covers the qualification testing of Type D19-XXX candidate spectrometer standards submitted by VHG Labs Inc. against the requirements of MIL-DTL-85694A. In general the candidate spectrometer standards met the requirements of the specification. A few specific data points were analyzed and waived based on alternative testing and analysis. As a result of this testing and analysis effort the VHG Lab Inc. D19-XXX series spectrometer standards have been qualified to the specification.

Since this qualification testing effort was the first time the full range of data required by MIL-DTL-85694A has been collected, analysis of the data has lead to the conclusion that several of the requirements of the specification are incorrect and must be revised. Specific recommended changes to the specification will be provided to all interested parties and NAVAIR will begin the process to officially revise MIL-DTL-85694A.

LIST OF ACRONYMS/ABBREVIATIONS

Joint Oil Analysis Program.....	JOAP
Standard Reference Material.....	SRM
Joint Oil Analysis Program-Coordinating Group	JOAP-CG
Rotrode Atomic Emission Spectroscopy	Rotrode-AES
Inductively Coupled Plasma Atomic Emission Spectroscopy.....	ICP-AES

Joint Oil Analysis Program Spectrometer Standards VHG Labs Inc. Qualification Report For D19-XXX Series Standards

1.0 BACKGROUND

The Joint Oil Analysis Program recently revised the specification for their spectrometric oil standards in order to incorporate an ICP-AES test method and to transition to commercially manufactured spectrometric oil standards. Previously a Rotrode-AES test method was the only elemental test method used to verify the quality of the spectrometric oil standards. The Rotrode-AES test method was a labor and time intensive process that did not exhibit a high level of accuracy and repeatability. The JOAP wanted to incorporate an ICP-AES test method in order to obtain a more accurate determination of the true concentration of the elements in the spectrometric oil standards and to reduce the cost and time needed to conduct quality assurance. Both the Rotrode-AES and ICP-AES test methods are now part of the qualification inspection in order to ensure that the formulation of the commercially manufactured spectrometric oil standards is correct.

The JOAP D19 reference series has historically consisted of true gravimetric spectrometric oil standards. The formulation of the JOAP D19 reference series has remained constant throughout the history of the program in order to ensure the accuracy of the aeronautical and non-aeronautical limits in the JOAP Manual (NAVAIR 17-15-50.3 and 17-15-50.4). The importance of maintaining the formulation of the JOAP D19 reference series provided another reason for the JOAP to transition to commercially manufactured spectrometric oil standards. Commercially manufactured spectrometric oil standards are generally traceable to NIST Standard Reference Materials (SRM). This traceability further ensures that the formulation of the commercially manufactured spectrometric oil standards is correct.

VHG Labs Inc. submitted Type D19-XXX series spectrometric oil standards that were manufactured to meet the requirements of MIL-DTL-85694A and are traceable to NIST SRMs. Testing was accomplished at the NAVAIR Patuxent River MD and Pensacola FL test facilities in accordance with the latest revision of MIL-DTL-85694A.

2.0 OBJECTIVE

The objective of this testing is to qualify a commercial vendor, VHG Labs Inc, for the production of blended spectrometric oil standards that are used in calibrating and verifying the calibration of spectrometers used in spectrometric analysis of metallic elements found in oils and other fluids.

3.0 APPROACH

The test requirements and methods for qualification inspection are specified in Tables I through III.

TABLE I. Physical Property Requirements and Test Methods.

Physical property	Requirement	Test Method
Density at 60 °F	report	ASTM D4052
Viscosity (mm ² /s at 100 °C)	16.3 min., 21.9 max.	ASTM D445
Viscosity Index, Min	85	ASTM D2270
Trace Sediment	0.005 ml/100 mL Oil max.	ASTM D2273
Pour point	-12 °C max.	ASTM D97
Flash point, minimum (COC)	243 °C	ASTM D92

The density, viscosity, viscosity index, trace sediment, pour point, and flash point test methods were conducted in accordance with the corresponding ASTM test method.

The Rotrode-AES test method was conducted in accordance with ASTM D6595. Testing of candidate spectrometric oil standards was done as a statistical analysis and performed on the data generated by the spectrometric analysis to provide an accuracy index and repeatability index for each element involved. The results were obtained using JOAP-CG approved Spectroil M/N spectrometers. The testing consisted of multiple replicates of each candidate spectrometric standard.

The ICP-AES test method was conducted in accordance with ASTM D5185. Testing of spectrometric oil standards was done as a statistical analysis and performed on the data generated by the spectrometric analysis to provide an accuracy index for each element involved. The results were obtained using JOAP-CG approved ICP spectrometers. The ICP spectrometer was calibrated from 0 ppm to 10 ppm using appropriate reference spectrometric standards. Candidate spectrometric oil standards were diluted on a weight-by-weight basis with a suitable ICP solvent. Candidate spectrometric oil standards in concentrations 5 ppm, 10 ppm, 30 ppm, and 50 ppm were diluted tenfold while candidate standards in concentrations 80 ppm, 100 ppm, 120 ppm, 300 ppm, 500 ppm, 700 ppm, and 900 ppm were diluted to 5 ppm. The testing consisted of five replicates of each candidate spectrometric standard.

4.0 DISCUSSION

This section details the analysis of the data according to the requirements published in the specification, MIL-DTL-85694A. An explanation of instrumental limitation and anomalies are provided in the event that the requirements are not met. Specification requirements that are not met will be subject to revision due to factors that influenced some chemical and physical properties that were not taken into consideration during the development of the specification.

TABLE II. Rotrode-AES (ASTM D6595) Accuracy and Repeatability Requirements

Element	Accuracy Index (AI)												Repeatability Index (RI)											
	0	5	10	30	50	80	100	120	300	500	700	900	0	5	10	30	50	80	100	120	300	500	700	900
Al	0.6	0.9	2.1	3.3	4.5	5.7	7.5	9.3	33.0	45.0	57.0	69.0	0.5	0.6	0.7	1.5	2.5	3.5	5.0	8.0	15.0	25.0	35.0	45.0
Cr	0.6	0.9	2.1	3.3	4.5	5.7	7.2	8.7	33.0	45.0	57.0	69.0	0.5	0.6	0.7	1.5	2.5	3.5	5.0	8.0	15.0	25.0	35.0	45.0
Cu	0.8	1.2	2.1	4.5	7.1	9.6	13.7	17.7	45.0	70.0	95.0	120.0	0.5	0.7	1.0	2.7	4.5	6.5	9.0	11.0	27.0	45.0	63.0	81.0
Fe	0.8	1.2	2.1	3.9	6.3	8.7	12.0	15.3	39.0	63.0	87.0	111.0	0.5	0.6	0.9	2.4	4.0	6.0	8.0	10.0	24.0	40.0	56.0	72.0
Pb	1.2	1.5	2.1	3.3	5.0	6.6	9.2	11.7	33.0	50.0	67.0	84.0	0.9	1.0	1.0	2.0	3.1	4.5	6.0	9.0	18.0	30.0	42.0	54.0
Mg	0.8	1.2	2.1	4.5	7.1	9.6	13.7	17.7	45.0	70.0	95.0	120.0	0.5	1.0	1.6	4.8	8.0	8.5	9.0	11.0	27.0	45.0	63.0	81.0
Ni	0.6	0.9	2.1	3.3	4.5	5.7	7.5	9.3	33.0	45.0	57.0	69.0	0.5	0.6	0.7	1.5	2.5	3.5	5.0	8.0	15.0	25.0	35.0	45.0
Si	0.6	0.9	2.1	3.3	4.5	5.7	7.5	9.3	33.0	45.0	57.0	69.0	0.5	0.6	0.7	1.5	2.5	3.5	5.0	8.0	15.0	25.0	35.0	45.0
Ag	1.0	1.8	2.1	3.9	6.3	8.7	12.0	15.3	39.0	63.0	87.0	111.0	0.5	0.6	0.9	2.4	4.0	6.0	8.0	10.0	24.0	40.0	56.0	72.0
Na	0.8	2.0	3.3	8.7	14.1	19.5	28.5	37.5	87.0	140.0	193.0	246.0	0.5	1.0	1.6	4.8	8.0	12.0	16.0	20.0	48.0	80.0	112.0	144.0
Sn	1.2	1.5	2.1	3.3	5.0	6.6	9.2	11.7	33.0	50.0	67.0	84.0	0.5	1.0	1.0	2.0	3.1	4.5	6.0	9.0	18.0	30.0	42.0	54.0
Ti	0.6	0.9	2.1	3.3	4.7	6.0	8.7	11.4	33.0	47.0	61.0	75.0	0.5	0.6	0.7	1.8	3.0	4.3	6.0	9.0	18.0	30.0	42.0	54.0
B	0.6	0.9	2.1	3.3	4.7	6.0	8.7	11.4	33.0	47.0	61.0	75.0	0.5	0.6	0.7	1.8	3.0	4.3	6.0	9.0	18.0	30.0	42.0	54.0
Mo	0.8	1.2	2.1	3.9	6.3	8.7	12.0	15.3	39.0	63.0	87.0	111.0	0.5	0.6	0.9	2.4	4.0	5.5	8.0	10.0	24.0	40.0	56.0	72.0
Zn	0.8	1.5	2.4	6.2	9.9	13.7	19.5	25.4	62.0	99.0	136.0	173.0	0.5	0.8	1.3	3.6	6.0	9.0	12.0	18.0	36.0	60.0	84.0	108.0
Ba	0.8	1.2	2.1	3.9	6.3	8.7	12.0	15.3	39.0	63.0	87.0	111.0	0.5	0.6	0.7	1.8	3.0	4.5	6.0	9.0	18.0	30.0	42.0	54.0
Cd	0.8	1.2	2.1	3.9	6.3	8.7	12.0	15.3	39.0	63.0	87.0	111.0	0.5	0.6	0.7	1.8	3.0	4.5	6.0	9.0	18.0	30.0	42.0	54.0
Mn	0.8	1.2	2.1	3.9	6.3	8.7	12.0	15.3	39.0	63.0	87.0	111.0	0.5	0.6	0.7	1.8	3.0	4.5	6.0	9.0	18.0	30.0	42.0	54.0
V	0.8	1.2	2.1	3.9	6.3	8.7	12.0	15.3	39.0	63.0	87.0	111.0	0.8	0.6	0.9	1.8	3.0	4.5	6.0	9.0	18.0	30.0	42.0	54.0
Ca	0.5	1.2	1.5	3.3	5.5	8.3	10.0	12.6	32.0	53.0	73.9	95.0	0.5	0.6	0.8	1.8	2.8	3.5	3.8	4.1	10.0	20.0	29.8	40.0
P	4.4	6.0	7.5	15.5	18.2	19.4	20.0	21.6	35.0	60.0	82.3	105.0	1.1	1.5	1.9	5.5	7.5	9.3	10.0	12.5	15.0	25.0	32.6	40.0

Note: The column headings, 0, 5, 10, 30, 50, 80, 100, 120, 300, 500, 700 and 900 denote the nominal concentration in parts per million (ppm).

TABLE III. ICP-AES (ASTM D5185) Accuracy Requirements

	Type D19-0	Type D19-XXX and D21-XXX											Type D12-XXX						Type D3-100
Element	0	5	10	30	50	80	100	120	300	500	700	900	5	10	30	50	100	300	100
Al	1.0	1.4	1.8	2.9	3.5	4.3	4.7	5.1	7.4	9.1	10.4	11.5	1.4	1.8	2.9	3.5	4.7	7.4	-----
Cr	1.0	0.6	1.0	2.2	3.2	4.5	5.4	6.2	12.3	18.0	23.1	27.9	0.6	1.0	2.2	3.2	5.4	12.3	-----
Cu	1.0	0.5	1.0	2.7	4.2	6.5	7.9	9.4	21.5	34.3	46.6	58.6	0.5	1.0	2.7	4.2	7.9	21.5	-----
Fe	1.0	0.5	0.8	2.0	3.0	4.3	5.2	6.0	12.5	18.8	24.5	30.0	0.5	0.8	2.0	3.0	5.2	12.5	-----
Pb	1.0	2.7	3.3	4.8	5.6	6.5	7.0	7.4	9.9	11.7	13.0	14.1	2.7	3.3	4.8	5.6	7.0	9.9	-----
Mg	1.0	0.6	1.2	3.0	4.6	6.9	8.4	9.8	21.6	33.5	44.8	55.6	0.6	1.2	3.0	4.6	8.4	21.6	-----
Ni	1.0	1.1	1.6	2.8	3.5	4.5	5.0	5.4	8.5	10.9	12.9	14.6	1.1	1.6	2.8	3.5	5.0	8.5	-----
Si	1.0	2.0	2.4	3.1	3.6	4.1	4.3	4.5	5.7	6.5	7.1	7.6	2.0	2.4	3.1	3.6	4.3	5.7	-----
Ag	1.0	0.6	1.0	2.5	3.9	5.7	6.9	8.0	17.1	26.1	34.5	42.5	0.6	1.0	2.5	3.9	6.9	17.1	-----
Na	1.0	1.4	2.2	4.6	6.5	8.8	10.2	11.5	21.1	29.6	37.0	43.7	1.4	2.2	4.6	6.5	10.2	21.1	-----
Sn	1.0	3.2	3.5	4.3	4.7	5.1	5.3	5.4	6.3	6.9	7.3	7.6	3.2	3.5	4.3	4.7	5.3	6.3	-----
Ti	1.0	1.0	1.3	1.9	2.3	2.7	3.0	3.2	4.5	5.4	6.1	6.7	0.9 ^A	1.2 ^A	1.8 ^A	2.2 ^A	2.9 ^A	4.3 ^A	-----
B	1.0	1.3	2.6	7.8	13.0	20.8	26.0	31.2	78.0	130.0	182.0	234.0	-----	-----	-----	-----	-----	-----	29.9 ^B
Mo	1.0	0.9	1.5	3.1	4.5	6.2	7.3	8.3	15.7	22.5	28.4	33.9	-----	-----	-----	-----	-----	-----	6.5 ^C
Zn	1.0	0.6	1.1	3.0	4.7	7.1	8.6	10.1	22.7	35.6	47.8	59.7	-----	-----	-----	-----	-----	-----	8.6
Ba	1.0	0.7	1.1	2.3	3.2	4.3	5.0	5.7	10.4	14.5	18.1	21.4	-----	-----	-----	-----	-----	-----	-----
Cd	1.0	0.8	1.5	4.5	7.5	12.0	15.0	18.0	45.0	75.0	105.0	135.0	-----	-----	-----	-----	-----	-----	-----
Mn	1.0	0.5	1.0	3.0	5.0	3.0	10.0	5.0	30.0	50.0	50.0	90.0	-----	-----	-----	-----	-----	-----	-----
V	1.0	0.3	0.6	1.8	3.1	4.9	6.1	7.3	18.3	30.5	42.7	54.9	-----	-----	-----	-----	-----	-----	-----
Ca	1.0	0.5	1.0	3.0	5.0	8.0	10.0	12.0	30.0	50.0	19.2	90.0	-----	-----	-----	-----	-----	-----	-----
P	1.0	3.3	4.9	9.3	12.6	16.5	18.8	20.9	35.5	47.8	58.1	67.2	-----	-----	-----	-----	-----	-----	-----

Note: The column headings, 0, 5, 10, 30, 50, 80, 100, 120, 300, 500, 700, and 900 denote the nominal concentration in parts per million (ppm).

A - Ti concentration = 0.92X, where X = nominal concentration of spectrometric oil standard in ppm.

B - B concentration = 1.15X, where X = nominal concentration of spectrometric oil standard in ppm.

C - Mo concentration = 0.85X, where X = nominal concentration of spectrometric oil standard in ppm.

4.1 Type D19-XXX Series

All of the Type D19-XXX spectrometric oil standards submitted by the candidate met the requirements for density at 60°F (ASTM D4052), viscosity index (ASTM 2270), trace sediment (ASTM D2273), and pour point (ASTM D97).

All of the Type D19-XXX spectrometric oil standards with the exception of D19-900 met the requirement for viscosity at 100°C (ASTM D445). The high concentration of the metallo-organic concentrates in D19-900 has an effect on the viscosity at 100°C which resulted in the requirement for viscosity at 100°C not being met. The significance of the effect was not taken into consideration during the development of the original specification requirements therefore the viscosity at 100°C requirement for D19-900 will require revision. At this time we are waiving the viscosity at 100°C requirement for D19-900.

The flash point (ASTM D92) requirements were met only for the D19-5, D19-10, and D19-30 spectrometric oil standards. All of the other Type D19-XXX spectrometric oil standards (D19-50 to D19-900) did not meet the requirement for flash point (ASTM D92). The high concentration of the metallo-organic concentrates in these standards has an effect on the flash point which resulted in the requirement for flash point not being met. The significance of the effect was not taken into consideration during the development of the original specification requirements therefore the flash point requirement will require revision. At this time we are waiving the flash point requirement for D19-50, D19-80, D19-100, D19-120, D19-300, D19-500, D19-700, and D19-900.

TABLE IV. Candidate Type D19-XXX Physical Property Results

TEST	METHOD	UNITS	SAMPLES										
			D19-5	D19-10	D19-30	D19-50	D19-80	D19-100	D19-120	D19-300	D19-500	D19-700	D19-900
Density at 60°F	ASTM D4052	kg/L	0.8839	0.8823	0.8841	0.8852	0.8865	0.8884	0.8893	0.8996	0.9114	0.9232	0.9351
Viscosity at 100°C	ASTM D445	cSt	19.1	19.1	19.1	19.1	19.2	19.2	19.2	19.4	19.9	20.7	22.1
Viscosity @ 40°C	ASTM D445	cSt	220.6	220.7	221.2	221.7	221.7	222.3	225.5	225.8	234.3	250.7	278.2
Viscosity Index	ASTM D2270	No Units	97	97	97	97	98	97	96	97	97	97	96
Pour Point	ASTM D97	°C	-15	-18	-18	-18	-15	-15	-15	-15	-18	-21	-21
Flash Point	ASTM D92	°C	268	256	254	240	234	215	220	174	153	142	110
Water & Sediment	ASTM D2273	%	0	0	0	0	0	0	0	0	0	0.002	0.003

NOTE: Red numbers indicate the requirement not being met

Most of the Type D19-XXX spectrometric oil standards originally did not meet the requirement for Rotrode-AES (ASTM D6595). Following standard JOAP quality assurance procedure, the candidate standards with concentrations greater than 50 ppm were subsequently diluted to 50 ppm using JOAP D19-0 and retested. Upon retesting, a significant number of the candidate standards still did not meet the Rotrode-AES requirements.

TABLE V. Candidate Type D19-XXX Rotrode-AES Original Results

TEST	METHOD	UNITS	SAMPLES																							
			D19-5		D19-10		D19-30		D19-50		D19-80		D19-100		D19-120		D19-300		D19-500		D19-700		D19-900			
Trace Metal:	ASTM D6595	ppm	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev		
Al			5.7	0.2	11.1	0.4	33.3	1.0	53.4	1.8	87.9	2.6	105.2	3.0	125.6	4.6	304.3	12.9	507.2	29.5	729.9	67.3	950.3	68.3		
Cr			5.6	0.3	11.5	0.5	33.7	1.6	54.4	3.1	89.9	3.1	113.7	3.7	137.9	4.4	333.1	20.8	529.5	40.3	781.4	87.4	999.3	53.7		
Cu			5.6	0.2	11.0	0.3	34.0	0.6	56.7	1.2	90.1	2.3	105.9	7.6	135.7	11.7	291.2	15.1	525.8	70.2	826.8	73.9	1005.5	137.6		
Fe			5.4	0.2	10.3	0.5	33.5	1.0	55.7	2.2	88.3	2.8	105.3	6.6	131.0	7.0	295.0	29.1	514.5	58.7	808.3	102.1	1037.8	103.6		
Pb			5.4	0.3	10.4	0.4	33.0	0.6	55.2	1.2	88.1	1.7	107.0	4.2	131.4	4.9	312.0	20.1	544.0	37.5	794.9	71.5	1024.3	51.2		
Mg			5.5	0.2	10.4	0.6	33.3	1.4	56.1	3.1	88.8	3.9	105.4	8.9	134.0	10.8	306.7	10.8	527.5	19.2	773.4	39.7	965.9	29.4		
Ni			5.8	0.1	11.3	0.2	33.1	0.6	54.9	1.1	88.1	1.6	108.4	3.0	133.5	3.3	310.3	25.5	512.6	48.2	769.3	105.0	1004.0	75.2		
Si			6.4	0.3	11.4	0.7	34.9	0.9	58.7	3.4	90.0	2.3	107.1	3.6	131.7	6.3	308.3	18.2	536.5	46.7	812.3	77.9	1029.2	66.9		
Ag			5.5	0.2	10.8	0.3	34.0	0.5	55.5	1.8	90.8	3.5	106.6	9.7	135.8	13.3	298.9	23.0	526.7	67.6	839.5	74.7	1059.2	129.1		
Na			5.7	0.4	11.6	0.7	34.1	2.3	56.7	4.9	92.3	4.5	113.5	8.8	155.6	14.4	387.9	39.2	540.5	25.9	748.8	39.4	889.5	34.9		
Sn			6.0	0.6	11.0	0.6	33.2	0.8	55.2	1.3	88.0	1.6	105.8	3.4	129.6	3.3	316.5	11.0	541.1	20.6	789.5	39.8	994.3	27.3		
Ti			5.4	0.2	10.5	0.5	33.4	1.4	56.0	2.8	88.0	3.8	104.1	8.0	130.4	10.3	288.6	39.4	514.4	62.2	804.0	117.7	1030.9	91.9		
B			5.1	0.2	9.4	0.6	29.8	0.9	50.2	1.9	80.4	2.3	95.6	2.1	120.8	6.4	291.7	11.6	513.9	33.7	776.4	56.2	996.7	56.1		
Mo			5.6	0.3	10.5	0.6	34.2	1.8	56.6	4.1	91.4	4.3	104.8	9.0	131.3	10.5	284.6	40.9	523.1	62.2	803.8	84.2	1001.1	80.6		
Zn			5.0	0.3	8.7	0.8	32.9	3.7	56.2	10.9	90.5	10.5	103.0	10.8	137.2	9.1	326.5	29.6	587.0	24.9	825.5	37.0	1024.9	26.6		
Ba			6.4	0.7	10.8	0.7	33.0	1.0	52.7	2.3	87.8	2.4	102.2	4.5	124.8	8.5	278.1	17.8	515.4	59.8	796.2	96.6	1006.5	99.7		
Cd			5.6	0.4	10.2	0.9	34.2	1.7	56.3	4.1	90.8	4.3	107.7	8.4	133.1	8.6	299.0	28.4	579.4	59.2	847.9	50.1	1033.6	68.5		
Mn			5.6	0.1	10.7	0.4	32.7	1.1	54.7	2.0	87.0	2.8	105.6	6.2	131.1	5.6	296.4	21.5	530.6	42.6	810.7	79.5	1050.8	68.6		
V			5.6	0.3	11.3	0.6	34.4	1.4	56.6	2.5	91.6	3.2	114.8	4.0	134.8	8.2	339.8	10.2	567.0	27.9	819.0	40.8	998.4	49.2		

NOTE: Obtained using R219 reference standards. Red numbers indicate the requirement not being met

TABLE VI. Candidate Type D19-XXX Rotrode-AES Retest Results

TEST	METHOD	UNITS	SAMPLES																							
			D19-5		D19-10		D19-30		D19-50		D19-80		D19-100		D19-120		D19-300		D19-500		D19-700		D19-900			
Trace Metal:	ASTM D6595	ppm	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev
Al											52.1	1.9	51.9	1.7	53.5	1.6	52.9	1.6	52.7	1.9	52.5	2.0	52.4	1.6		
Cr											54.5	1.7	54.4	1.6	55.9	2.1	56.2	2.0	55.8	2.7	57.1	1.9	58.0	1.9		
Cu											54.8	1.7	54.9	1.7	56.5	0.6	55.7	2.3	56.5	0.8	55.2	1.9	55.5	1.5		
Fe											55.5	2.3	55.5	2.0	55.8	1.9	55.4	2.5	55.7	1.3	54.1	2.7	54.7	2.3		
Pb											54.5	1.6	54.7	1.1	56.0	1.1	55.6	1.9	56.1	1.0	55.6	2.0	56.0	1.7		
Mg											55.4	2.7	55.6	2.7	56.0	2.3	54.8	3.6	56.0	1.8	53.6	3.3	54.1	2.8		
Ni											54.7	1.1	54.4	1.3	55.9	0.9	56.4	1.1	56.4	0.9	56.9	1.6	57.6	1.0		
Si											56.0	1.9	57.1	1.6	57.8	1.6	57.7	1.9	57.9	1.8	56.9	2.5	57.0	2.2		
Ag											54.1	2.1	54.1	2.0	55.8	1.2	54.3	3.0	55.5	1.4	53.2	2.6	53.4	2.0		
Na											55.0	2.3	55.9	2.3	56.1	3.9	57.5	4.0	57.1	3.3	58.0	2.5	59.2	2.1		
Sn											54.9	1.3	55.0	1.4	55.8	0.8	55.2	2.0	56.0	0.8	55.2	2.0	55.9	1.5		
Ti											54.3	2.1	54.0	2.6	56.0	2.6	54.6	3.9	56.4	2.5	53.5	3.3	53.7	2.6		
B											50.4	1.8	50.5	1.5	49.3	1.9	49.7	2.0	49.8	1.2	49.3	1.6	49.9	1.9		
Mo											54.3	3.5	55.3	3.4	54.1	3.8	52.3	4.7	54.5	4.1	50.8	3.6	51.1	3.9		
Zn											49.6	6.0	49.6	4.6	53.4	7.4	52.0	8.2	53.6	7.7	47.9	6.4	49.1	7.5		
Ba											50.2	2.7	50.2	2.0	52.1	1.9	52.0	1.7	50.9	1.7	50.4	2.7	51.0	2.7		
Cd											52.7	2.8	54.5	2.5	53.5	3.0	52.3	3.4	52.9	2.7	50.2	2.4	50.3	3.1		
Mn											54.9	2.2	54.5	2.3	55.3	1.3	54.7	3.0	55.3	1.2	54.2	3.1	55.1	2.1		
V											57.8	1.9	58.1	1.7	57.2	2.9	57.8	1.7	57.9	1.7	58.0	1.6	58.5	1.3		

NOTE: Obtained using R219 reference standards. Red numbers indicate the requirement not being met

One possible explanation for the Rotrode-AES requirements not being met is that the existing JOAP D19 reference series, commonly referred to as R219, was nearing expiration and thus might not be suitable for use. The NAVAIR Patuxent River MD and Pensacola FL test facilities sought the assistance of laboratories at Spectro, Inc and VHG Labs, Inc to collect additional data which would be used by NAVAIR employees to corroborate the usability of R219.

It was determined that R219 was not suitable for use due to significant deviation from the expected concentrations of several elements. The exact cause of the significant deviation cannot be determined and was not the primary interest of any of the parties involved. However all parties concluded that issues associated with the approaching expiration and possible formulation drift most likely caused the deviation.

The NAVAIR Pensacola FL facility was unable to manufacture a new reference D19 series due to ongoing material and staffing shortages. Therefore, the NAVAIR test facilities consulted with the necessary parties and decided to obtain suitable third party 19-element reference standards in order to complete the Rotrode-AES qualification testing. Subsequent Rotrode-AES tests were

conducted using the third party reference standard. All of the candidate Type D19-XXX spectrometric oil standards met the requirements for Rotrode-AES (ASTM D6595).

TABLE VII. Candidate Type D19-XXX Rotrode-AES Final Results

TEST	METHOD	UNITS	SAMPLES																							
			D19-5		D19-10		D19-30		D19-50		D19-80		D19-100		D19-120		D19-300		D19-500		D19-700		D19-900			
Trace Metal:	ASTM D6595	ppm	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev
Al			5.2	0.1	10.2	0.1	30.2	0.2	49.1	0.4	46.8	0.9	98.0	2.7	47.5	0.5	47.6	1.0	47.7	1.5	47.4	0.2	48.2	1.1		
Cr			5.4	0.1	10.7	0.0	32.6	0.2	48.8	0.6	49.1	0.9	101.2	4.0	49.6	0.4	49.9	1.3	50.0	1.6	49.6	0.6	48.4	0.8		
Cu			5.2	0.1	10.5	0.2	31.3	0.1	52.9	1.4	51.6	1.4	96.6	3.6	52.8	0.5	52.2	1.1	52.7	2.8	52.1	1.3	51.8	1.4		
Fe			4.8	0.1	9.7	0.3	28.9	0.6	49.3	0.8	46.8	1.1	96.0	4.1	47.7	0.5	47.0	0.6	47.3	2.2	47.0	0.8	47.7	1.2		
Pb			4.8	0.4	9.9	0.3	29.8	0.7	49.9	0.6	48.3	1.1	97.2	2.9	49.1	0.4	48.7	0.7	48.5	2.0	48.3	0.9	48.6	1.1		
Mg			5.1	0.2	10.1	0.3	30.0	0.5	49.6	0.5	47.4	1.5	97.6	3.1	48.1	0.9	47.6	1.1	47.8	1.0	47.6	1.0	48.1	1.4		
Ni			5.1	0.1	10.2	0.2	30.6	0.1	51.5	1.0	49.9	0.9	101.0	3.2	50.5	0.4	50.2	0.8	50.4	1.4	50.3	1.0	50.5	1.2		
Si			5.4	0.1	10.6	0.3	30.9	0.3	53.5	0.6	51.6	0.7	101.7	3.7	52.4	1.3	51.1	0.6	51.6	2.5	50.9	0.9	51.6	1.0		
Ag			5.3	0.1	10.6	0.2	31.5	0.5	51.9	0.8	50.1	1.5	103.8	7.4	50.9	0.6	50.0	1.1	50.9	3.8	49.7	0.6	50.5	1.2		
Na			5.8	0.2	11.0	0.3	31.6	1.1	52.8	2.5	57.6	1.4	105.7	3.2	56.5	1.1	56.5	0.5	55.9	2.1	58.1	1.0	52.5	0.9		
Sn			4.9	0.5	9.8	0.8	29.8	0.8	49.6	0.6	47.9	1.2	97.0	2.7	48.6	0.4	48.1	1.0	48.2	1.6	47.9	1.0	48.4	1.2		
Ti			5.1	0.1	10.3	0.4	30.6	0.5	52.5	1.1	48.2	1.1	98.6	2.7	48.9	0.5	48.5	0.8	48.4	1.8	48.6	1.3	50.9	1.7		
B			5.0	0.1	10.1	0.3	30.4	0.7	51.2	0.9	49.4	0.4	100.7	3.0	50.0	0.3	50.0	0.5	50.3	2.1	50.1	0.8	49.6	0.7		
Mo			4.9	0.2	9.4	0.4	27.7	0.8	45.7	0.9	44.3	0.6	95.3	5.0	44.9	0.2	44.1	0.9	44.3	2.8	44.4	1.1	44.0	0.8		
Zn			4.9	0.0	9.6	0.6	28.6	1.5	45.0	1.0	45.6	0.5	96.9	2.4	45.6	0.7	45.1	2.6	45.0	5.2	44.1	1.3	44.2	0.6		
Ba			5.7	0.1	10.6	0.4	29.4	0.3	47.6	0.6	50.6	1.4	94.9	4.8	49.4	1.5	50.9	1.3	50.5	1.8	51.9	1.6	48.4	0.2		
Cd			5.2	0.1	10.3	0.3	31.2	0.8	50.0	0.4	48.3	0.1	102.7	4.8	48.9	0.4	48.7	1.0	48.7	2.5	48.3	1.0	48.8	0.5		
Mn			4.5	0.2	9.8	0.4	30.8	0.2	50.7	1.0	49.6	1.0	96.9	3.4	50.4	0.3	50.1	1.0	50.3	1.7	49.9	1.0	49.3	1.2		
V			5.0	0.1	9.7	0.2	29.3	0.3	50.0	1.2	49.0	0.8	98.7	1.5	49.6	0.2	49.8	0.9	49.8	1.2	49.9	1.0	49.6	1.0		

NOTE: Obtained using third party reference standard. Red numbers indicate the requirement not being met

The D19-30 spectrometric oil standards met the requirement for ICP-AES (ASTM D5185) while the D19-5, D19-10, D19-50, D19-80, D19-100, D19-120, D19-300, D19-500, D19-700, and D19-900 did not. Elevated sodium, boron, and vanadium levels were observed in the ICP-AES D19-5 data and not in the Rotrode-AES D19-5 data. This suggests the elevated ICP-AES readings are instrumental anomalies and the failures can be waived. Upon further analysis, it was determined that the accuracy listed in the specification did not accurately take into consideration the dilution factors. Therefore the dilution factors were removed from each sample and the ICP-AES data were reevaluated using the appropriate limits for the given concentration. Table XII lists for each element in the Type D19-XXX spectrometric oil standards the average ICP result without the dilution factor, the expected concentration, and the appropriate limit as originally published in the specification.

TABLE VIII. Candidate Type D19-XXX ICP-AES Results

TEST	METHOD	UNITS	SAMPLES											
			D19-5	D19-10	D19-30	D19-50	D19-80	D19-100	D19-120	D19-300	D19-500	D19-700	D19-900	
ICP	ASTM D5185	ppm	Mean	Mean	Mean	Mean	Mean	Mean	Mean	Mean	Mean	Mean	Mean	
Al			5.0	10.5	29.5	52.0	84.6	106.8	126.3	322.1	540.2	747.5	958.5	
Cr			4.7	9.8	28.9	49.9	79.0	100.5	116.1	308.2	507.7	665.3	923.7	
Cu			5.0	9.7	28.0	47.9	75.1	95.1	110.0	287.7	467.7	626.6	861.3	
Fe			5.2	9.8	28.0	47.9	75.6	96.7	110.9	295.5	486.9	633.9	886.8	
Pb			5.0	11.0	29.7	51.8	84.5	105.5	126.3	323.3	545.7	741.1	960.7	
Mg			5.2	10.1	29.1	49.2	77.7	99.0	114.9	300.4	497.7	658.7	903.4	
Ni			5.5	11.2	31.0	53.4	87.2	109.0	130.5	329.2	559.2	770.5	986.9	
Si			5.6	10.8	30.6	53.4	85.9	108.0	128.1	328.6	551.4	770.8	981.9	
Ag			5.1	10.9	30.3	53.9	87.0	107.9	130.0	323.8	542.8	766.8	968.4	
Na			6.9	12.9	31.9	56.8	93.3	116.3	142.2	355.8	606.1	855.8	1053.6	
Sn			5.0	9.9	28.6	49.1	77.8	100.6	114.8	310.7	513.6	663.1	931.9	
Ti			5.6	10.3	29.7	49.8	79.9	100.7	118.9	301.9	504.3	689.7	902.2	
B			6.6	10.9	30.3	51.5	84.7	96.4	127.6	241.8	373.6	742.1	923.0	
Mo			4.3	9.4	27.8	48.5	77.0	96.2	113.2	291.0	479.9	654.7	874.0	
Zn			4.9	9.3	27.3	47.0	74.2	94.3	109.2	287.4	478.0	627.3	872.0	
Ba			4.3	9.9	28.3	50.0	81.6	101.1	122.8	313.9	523.9	724.9	929.8	
Cd			4.6	9.5	28.4	49.5	78.4	100.7	115.7	308.7	506.9	668.3	930.3	
Mn			5.2	9.9	28.2	48.4	76.1	97.1	112.9	298.7	492.9	646.9	895.5	
V			5.5	10.7	29.9	51.3	81.9	102.3	121.2	311.3	516.9	704.1	936.1	

NOTE: Red numbers indicate the requirement not being met

All of the Type D19-XXX spectrometric oil standards with the exception of D19-5 met the requirement for ICP-AES (ASTM D5185) once the dilution factors were removed. The elevated levels for D19-5 are still viewed as an instrumental anomaly and the failure can be waived.

TABLE IX. Reevaluation of Candidate Type D19-XXX ICP-AES Results

TEST	METHOD	UNITS	SAMPLES											
			D19-5	D19-10	D19-30	D19-50	D19-80	D19-100	D19-120	D19-300	D19-500	D19-700	D19-900	
ICP	ASTM D5185	ppm	Avg / Exp / Limit	Avg / Exp / Limit	Avg / Exp / Limit	Avg / Exp / Limit	Avg / Exp / Limit	Avg / Exp / Limit	Avg / Exp / Limit	Avg / Exp / Limit	Avg / Exp / Limit	Avg / Exp / Limit	Avg / Exp / Limit	Avg / Exp / Limit
Al			0.50 / 0.50 / 0.14	1.1 / 1.0 / 0.3	2.9 / 2.9 / 0.8	5.3 / 5.1 / 1.4	5.1 / 4.8 / 1.4	5.2 / 4.8 / 1.3	5 / 4.8 / 1.3	6 / 5.6 / 1.6	5.5 / 5.1 / 1.4	5.7 / 5.4 / 1.5	5.1 / 4.6 / 1.3	
Cr			0.47 / 0.50 / 0.06	1.0 / 1.0 / 0.1	2.8 / 2.9 / 0.4	5.1 / 5.1 / 0.6	4.8 / 4.8 / 0.6	4.8 / 4.8 / 0.6	4.6 / 4.8 / 0.6	5.8 / 5.6 / 0.7	5.2 / 5.1 / 0.6	5.1 / 5.4 / 0.6	5.1 / 4.6 / 0.6	
Cu			0.50 / 0.50 / 0.05	1.0 / 1.0 / 0.1	2.7 / 2.9 / 0.3	4.9 / 5.1 / 0.5	4.5 / 4.8 / 0.5	4.6 / 4.8 / 0.5	4.4 / 4.8 / 0.5	5.4 / 5.6 / 0.6	4.7 / 5.1 / 0.5	4.8 / 5.4 / 0.5	4.8 / 4.6 / 0.5	
Fe			0.51 / 0.50 / 0.05	1.0 / 1.0 / 0.1	2.7 / 2.9 / 0.3	4.9 / 5.1 / 0.5	4.6 / 4.8 / 0.5	4.7 / 4.8 / 0.5	4.4 / 4.8 / 0.5	5.5 / 5.6 / 0.6	4.9 / 5.1 / 0.5	4.9 / 5.4 / 0.5	4.8 / 4.6 / 0.5	
Pb			0.50 / 0.50 / 0.27	1.1 / 1.0 / 0.5	2.9 / 2.9 / 1.6	5.3 / 5.1 / 2.8	5.1 / 4.8 / 2.6	5.1 / 4.8 / 2.6	5 / 4.8 / 2.6	6.1 / 5.6 / 3	5.5 / 5.1 / 2.7	5.7 / 5.4 / 2.9	5.1 / 4.6 / 2.5	
Mg			0.52 / 0.50 / 0.06	1.0 / 1.0 / 0.1	2.8 / 2.9 / 0.4	5.1 / 5.1 / 0.6	4.7 / 4.8 / 0.6	4.8 / 4.8 / 0.6	4.5 / 4.8 / 0.6	5.6 / 5.6 / 0.7	5.1 / 5.1 / 0.6	5 / 5.4 / 0.6	4.9 / 4.6 / 0.6	
Ni			0.55 / 0.50 / 0.11	1.1 / 1.0 / 0.2	3 / 2.9 / 0.6	5.5 / 5.1 / 1.1	5.3 / 4.8 / 1.1	5.3 / 4.8 / 1.1	5.2 / 4.8 / 1.1	6.2 / 5.6 / 1.2	5.7 / 5.1 / 1.1	5.9 / 5.4 / 1.2	5.3 / 4.6 / 1.1	
Si			0.56 / 0.50 / 0.20	1.1 / 1.0 / 0.4	3 / 2.9 / 1.2	5.5 / 5.1 / 2.1	5.2 / 4.8 / 1.9	5.2 / 4.8 / 1.9	5.1 / 4.8 / 1.9	6.2 / 5.6 / 2.3	5.6 / 5.1 / 2	5.9 / 5.4 / 2.1	5.4 / 4.6 / 1.8	
Ag			0.51 / 0.50 / 0.06	1.1 / 1.0 / 0.1	3 / 2.9 / 0.4	5.5 / 5.1 / 0.6	5.3 / 4.8 / 0.6	5.2 / 4.8 / 0.6	5.1 / 4.8 / 0.6	6.1 / 5.6 / 0.7	5.5 / 5.1 / 0.6	5.9 / 5.4 / 0.6	5 / 4.6 / 0.6	
Na			0.69 / 0.50 / 0.14	1.3 / 1.0 / 0.3	3.1 / 2.9 / 0.8	5.8 / 5.1 / 1.4	5.6 / 4.8 / 1.4	5.6 / 4.8 / 1.3	5.6 / 4.8 / 1.3	6.7 / 5.6 / 1.6	6.2 / 5.1 / 1.4	6.5 / 5.4 / 1.5	5.4 / 4.6 / 1.3	
Sn			0.49 / 0.50 / 0.32	1.0 / 1.0 / 0.6	2.8 / 2.9 / 1.9	5 / 5.1 / 3.3	4.7 / 4.8 / 3.1	4.9 / 4.8 / 3.1	4.5 / 4.8 / 3	5.8 / 5.6 / 3.6	5.2 / 5.1 / 3.3	5.1 / 5.4 / 3.4	4.9 / 4.6 / 3	
Ti			0.55 / 0.50 / 0.10	1.0 / 1.0 / 0.2	2.9 / 2.9 / 0.6	5.1 / 5.1 / 1	4.8 / 4.8 / 1	4.9 / 4.8 / 1	4.7 / 4.8 / 1	5.7 / 5.6 / 1.1	5.1 / 5.1 / 1	5.3 / 5.4 / 1.1	4.9 / 4.6 / 0.9	
B			0.65 / 0.50 / 0.13	1.1 / 1.0 / 0.3	3 / 2.9 / 0.8	5.3 / 5.1 / 1.3	5.1 / 4.8 / 1.3	4.7 / 4.8 / 1.3	5 / 4.8 / 1.2	4.5 / 5.6 / 1.5	3.8 / 5.1 / 1.3	5.7 / 5.4 / 1.4	5 / 4.6 / 1.2	
Mo			0.43 / 0.50 / 0.09	1.0 / 1.0 / 0.2	2.7 / 2.9 / 0.5	5 / 5.1 / 0.9	4.7 / 4.8 / 0.9	4.6 / 4.8 / 0.9	4.5 / 4.8 / 0.9	5.5 / 5.6 / 1	4.9 / 5.1 / 0.9	5 / 5.4 / 1	4.6 / 4.6 / 0.8	
Zn			0.48 / 0.50 / 0.06	1.0 / 1.0 / 0.1	2.7 / 2.9 / 0.4	4.8 / 5.1 / 0.6	4.5 / 4.8 / 0.6	4.5 / 4.8 / 0.6	4.3 / 4.8 / 0.6	5.4 / 5.6 / 0.7	4.9 / 5.1 / 0.6	4.8 / 5.4 / 0.6	4.6 / 4.6 / 0.6	
Ba			0.43 / 0.50 / 0.07	1.0 / 1.0 / 0.1	2.8 / 2.9 / 0.4	5.1 / 5.1 / 0.7	4.9 / 4.8 / 0.7	4.9 / 4.8 / 0.7	4.9 / 4.8 / 0.7	5.9 / 5.6 / 0.8	5.3 / 5.1 / 0.7	5.5 / 5.4 / 0.8	5.1 / 4.6 / 0.6	
Cd			0.46 / 0.50 / 0.08	1.0 / 1.0 / 0.2	2.8 / 2.9 / 0.5	5.1 / 5.1 / 0.8	4.7 / 4.8 / 0.8	4.9 / 4.8 / 0.8	4.6 / 4.8 / 0.8	5.8 / 5.6 / 0.9	5.1 / 5.1 / 0.8	5.1 / 5.4 / 0.9	4.9 / 4.6 / 0.7	
Mn			0.52 / 0.50 / 0.05	1.0 / 1.0 / 0.1	2.7 / 2.9 / 0.3	5 / 5.1 / 0.5	4.6 / 4.8 / 0.5	4.7 / 4.8 / 0.5	4.5 / 4.8 / 0.5	5.6 / 5.6 / 0.6	5 / 5.1 / 0.5	4.9 / 5.4 / 0.5	4.9 / 4.6 / 0.5	
V			0.55 / 0.50 / 0.03	1.1 / 1.0 / 0.1	2.9 / 2.9 / 0.2	5.3 / 5.1 / 0.3	5 / 4.8 / 0.3	4.9 / 4.8 / 0.3	4.8 / 4.8 / 0.3	5.8 / 5.6 / 0.3	5.2 / 5.1 / 0.3	5.4 / 5.4 / 0.3	5 / 4.6 / 0.3	

NOTE: Red numbers indicate the requirement not being met

The Type D19-XXX spectrometric oil standards submitted by the candidate satisfactorily passed the qualification inspection.

5.0 CONCLUSIONS

The following spectrometric oil standards submitted by VHG Labs Inc. have satisfactorily passed the qualification inspection and should be added to the qualified products list for MIL-DTL-85694A:

- Type D19-XXX (D19-5, D19-10, D19-30, D19-50, D19-80, D19-100, D19-120, D19-300, D19-500, D19-700, D19-900)

6.0 RECOMMENDATIONS

A number of issues associated with the specification requirements were brought to light as a result of the qualification testing. Several changes to the specification are therefore under consideration. Detailed recommended changes to MIL-DTL-85694A will be provided to all interested parties and the NAVAIR test facilities will begin the process to officially revise MIL-DTL-85694A.

REPORT DOCUMENTATION PAGE				Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing this collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden to Department of Defense, Washington Headquarters Services, Directorate for Information Operations and Reports (0704-0188), 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302. Respondents should be aware that notwithstanding any other provision of law, no person shall be subject to any penalty for failing to comply with a collection of information if it does not display a currently valid OMB control number. PLEASE DO NOT RETURN YOUR FORM TO THE ABOVE ADDRESS.					
1. REPORT DATE (DD-MM-YYYY) 11-04-2013		2. REPORT TYPE Technical		3. DATES COVERED (From - To) 08-20-2011 to 11-04-2013	
4. TITLE AND SUBTITLE Joint Oil Analysis Program Spectrometer Standards; VHG Labs Inc. Qualification Report for D19-XXX Series Standards				5a. CONTRACT NUMBER N/A	
				5b. GRANT NUMBER N/A	
				5c. PROGRAM ELEMENT NUMBER N/A	
6. AUTHOR(S) Peretich, Michael; Author Eldridge, Geoffrey.; Editor Krizovensky, John ; Editor Mearns, Douglas ; Editor				5d. PROJECT NUMBER N/A	
				5e. TASK NUMBER N/A	
				5f. WORK UNIT NUMBER N/A	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Naval Fuels & Lubricants Cross Functional Team 22229 Elmer Road Patuxent River, MD 20670				8. PERFORMING ORGANIZATION REPORT NUMBER NF&LCFT Report 441/13-015	
9. SPONSORING / MONITORING AGENCY NAME(S) AND ADDRESS(ES) Naval Air Systems Command NPRE Program 22347 Cedar Point Road Patuxent River MD 20670				10. SPONSOR/MONITOR'S ACRONYM(S) N/A	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S) N/A	
12. DISTRIBUTION / AVAILABILITY STATEMENT A Approved for public release; distribution is unlimited.					
13. SUPPLEMENTARY NOTES N/A					
14. ABSTRACT The Joint Oil Analysis Program recently revised the specification for their spectrometric oil standards in order to incorporate an ICP-AES (Inductively Coupled Plasma Atomic Emission Spectroscopy) test method and to transition to commercially manufactured spectrometric oil standards. Previously a Rotrode-AES (Atomic Emission Spectrometry) test method was the only elemental test method used to verify the quality of the spectrometric oil standards. The Rotrode-AES test method was a labor and time intensive process that did not exhibit a high level of accuracy and repeatability. The JOAP wanted to incorporate an ICP-AES test method in order to obtain a more accurate determination of the true concentration of the elements in the spectrometric oil standards and to reduce the cost and time needed to conduct quality assurance. Both the Rotrode-AES and ICP-AES test methods are now part of the qualification inspection in order to ensure that commercially manufactured spectrometric oil standards are identical to those that have been produced in-house by the JOAP. As a result this testing effort the VHG Lab Inc. Type D19-XXX series spectrometer standards are qualified to the MIL-DTL-85694A specification. Since this qualification testing effort was the first time the full range of data required by MIL-DTL-85694A has been collected, analysis of the data has lead to the conclusion that several of the requirements of the specification are incorrect and must be revised. Specific recommended changes to the specification will be provided to all interested parties and the NAVAIR test facilities will begin the process to officially revise MIL-DTL-85694A.					
15. SUBJECT TERMS Joint Oil Analysis Program, Spectrometer Standards					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT UNCLASSIFIED	b. ABSTRACT UNCLASSIFIED	c. THIS PAGE UNCLASSIFIED			Douglas F. Mearns
			Unclassified Unlimited	15	19b. TELEPHONE NUMBER (include area code) 301-757-3421